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CENTRAL INTELLIGENCE AGENCY

INFORMATION REPORT

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COUNTRY Rumania

SUBJECT Petroleum Research/Organizational Set-up

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1. "The Ministry of the Petroleum and Coal Industries (MIPIC) is responsible to the Council of Ministers. Its activities enter the natural gas (methane) industry as well as coal and petroleum. While the normal function of a ministry is the complete operation of a particular industrial sector, MIPIC is a sort of stepchild in that the petroleum industry - all oilfields, pipelines, refineries, etc. - is operated independent of the Ministry by Sovrompetrol. Thus the petroleum section of the Ministry has no functions vital to production, but is left to such activities as political work among personnel, collection of statistics, operation of technical schools, etc. In the opinion of many people, it exists primarily to mask the fact that petroleum, the country's most valuable resource, is being exploited by Soviet imperialism. Within the framework of this semi-purposelessness operates the Institut de Cercetări si Proectări (Institute of Research and Engineering Designs).

2. "The Ministry is headed by the Minister, an old-time Party member, a former laboratory assistant at the Româno-Americana refinery. Since he is not a technical specialist, he has two executive assistants, one heading up activities in petroleum, the other, coal. The assistant in charge of petroleum was Iancu.

3. "The Institute, although reorganized on an average of more than once a year since around 1946 when it began as the Institute of Chemistry and Technology with a broad field of chemical and mineral problems, had settled down by 1951 to two divisions: Research and Engineering Designs. The Director, Iordan Marcovici (formerly of Astra Româna) conducted actively both the technical and administrative work.

4. "The Engineering Design section, headed by a well-respected engineer named Heiman, had a staff of 20-30 architects and engineers, who prepared construction plans for facilities within the jurisdiction of the Ministry, such as coal mine buildings, housing units for petroleum workers, research equipment, etc. It was housed at the Institute's central offices at 23 Calea Grivitei.

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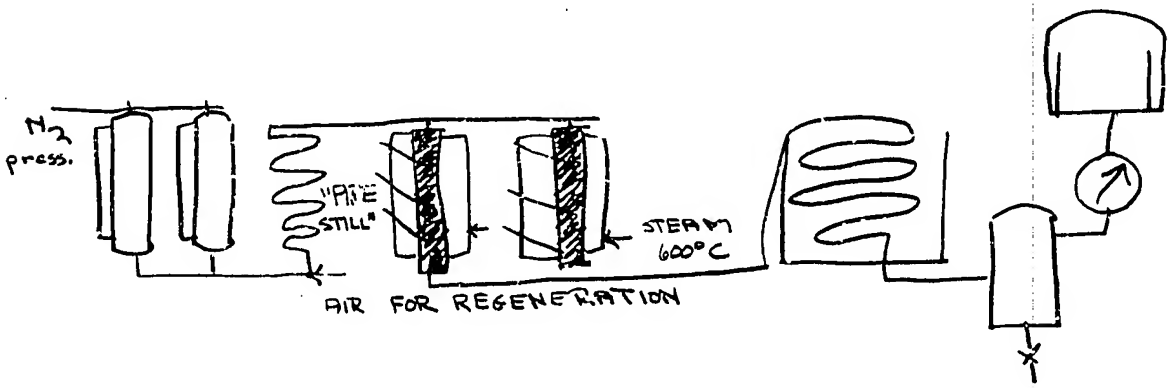
5. "The Research Section consisted of half-a-dozen cdd groups, working for the most part in laboratories loaned by other scientific institutions, such as the University, the Polytechnic Institute, the Geologic Institute, the Institute for Petroleum and Gases (a specialized technical faculty). The majority of these groups were led part-time by a professor of the host institution, and consisted furthur of 2-3 chemist-engineers and a laboratory technician. In August, 1951 the Research Laboratory was brought together at the remodeled factory, 'Tehnomarina' at Sos Vitan 112, formerly used for the production of greases and polishes. Its technical chief in early 1952 was Dr. Gheorghe Albescu (assistant superintendent at the Concordia-Vega refinery during the late 1930's); its administrative chief was Marcel Gheorghiu. Technical chief for work on coal was to be Dr. Blum of the Polytechnic Institute.

6. "Projects under way during 1951-2 were initiated in various ways. An assignment from the Ministerial level, reflecting a request from another Ministry or the Council of Ministers, naturally gave considerable impetus and priority to a project, especially for as long as signs of sustained interest were felt. Examples, catalytic cracking, toluene from naphtha. Other projects arose out of need, which made itself felt in a less formal way. For instance, paraffin oxidation began due to the universally felt need of fatty acids for soap. Processes mentioned by implication in the State Plans were on future agendas. The majority of projects went on through a single virtue: any equipment required could be improvised from ordinary iron with an old lathe and by welding. Since such projects presented the least problems, they occupied much of the personnel and dragged on until their justification had faded from the memory of almost everyone.

7. "Data on the various projects, as memory serves, follows. Parentheses give the names of personnel responsible, and their former industrial connections, if any are known.

8. "The most urgent project at the end of 1951 was the production of toluene from naphtha, which was being pressed by the Army and the Ministry of the Chemical and Metalurgical Industry, who would be responsible for the production of TNT. As the urgency heightened a schedule was set: pilot plant on stream 1 April 1952; construction of industrial units to begin December 1952. Verification of progress on behalf of the parties mentioned above was done by a Dr. Cismaru. However, overall planning was so deficient that responsibility for design of the industrial plant was apparently still unassigned when the pilot started up. As a result, the objectives of the pilot studies were never pinned down specifically. No evidence was to be seen that other aspects of the problem were being studied in other laboratories, or even that concrete technical thinking was going on elsewhere.

9. "The hydroforming pilot plant for toluene preparation consisted specifically of two twenty-liter reactors made from six-inch pipe, and auxiliary apparatus as shown in the following sketch:



10. "A catalyst of chromium oxide on alumina was used. Feed from nitrogen-pressured feed tanks fitted with calibrated sight glasses, was passed through a ½" pipe some 20-30' long, zig-zag in shape and heated by gases from an oil flame (in other words, a miniature pipe-still) before entering the reactors. The latter were heated by steam super-heated to about 600°C in another 'pipe-still'. Temperatures were determined with the help of two simple milliameter-pyrometers through multiple-contact switches. Reaction products were handled conventionally by condensers, wet-test meters, gas holders, etc.

11. "Catalyst was prepared by co-precipitation. Acidified potassium dichromate was reduced to chromic state by heating with alcohol; alum solution was added, and then ammonium hydroxide. The precipitate was washed repeatedly, filtered, and dried. The material was then powdered, pelleted in pill machines, and activated by slow heating to 500°C. Even after the pilot plant went on stream, no catalysts had been made based on molybdena, or by other methods, such as impregnation of commercial alumina.

12. "The purpose of the pilot study was apparently considered to be primarily verification of data from laboratory, rather than the collection of facts needed for engineering design of larger units. In general the laboratory data, obtained from 100cc of catalyst immersed in a lead bath, were considered to approximate values reported in the US literature for commercial operation: at feed rates of .1 to .3 vol/vol/hr, and cycles up to 2 hours, liquid recoveries of 60 to 80% and aromatic concentrations up to 65% were shown at reaction temperatures of 500-535°C. Conversion of methyl cyclohexane to toluene was apparently quite complete. Since the feed used had 10-20% C_6 and 10% or more of C_8, and since not even laboratory columns of more than 10-15 theoretical plates were available for testing purposes, no detailed study of the reaction was made.

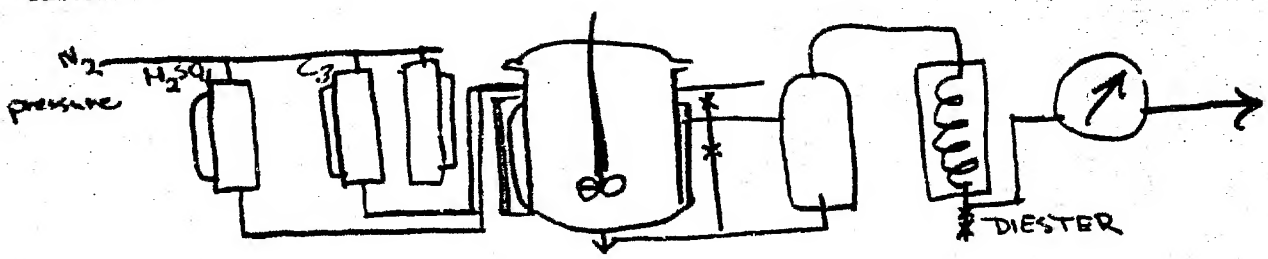
13. "Any possibility of collecting data on which to base an industrial design was rendered extremely remote by several limitations of the pilot plant. First, the mode of heating the reactors was too unrealistic. Thermal gradients, particularly during regeneration, would be completely different in larger, truly adiabatic reactors. Then feed inlet temperatures and feed rates could not be controlled as well as was needed for reliable results.

14. "It may be concluded that the most significant result of this pilot project, ambitious as it was considering that the Rumanian petroleum industry had previously done almost no development work of this type, was to show the immaturity of some of its conceptions. The 'pipe-still' type preheaters, a mimicry of industrial scale design, even with constant attention could not give controllable feed temperatures. The thermocouples, located in 3 wells projecting diagonally through the reactor walls, did not permit one to follow shifting temperatures, particularly the hot-spot during reactivation. This was one cause leading to the total sintering of the first catalyst charge into brick-red lumps half their previous diameter. The 'pipe-stills', being of ordinary soft steel pipe, burned through in the direct fire of the furnace after about a week of operation. A measure of the type of planning given this project is the fact that no distillation equipment, either laboratory or pilot plant, was suggested in connection with the actual isolation of toluene from saturates to bring it to nitration grade. While feed might conceivably be prepared with existing equipment and know-how, no such assumption could be made regarding the special distillations, azeotropic or extractive, needed by the product.

15. "A second important project was the preparation of isopropyl alcohol from refinery gases. Although studied intermittently in Rumania since 1935 or so, this process had never been worked out to the point of producing a commercial grade of product. Due to a need for acetone in the synthesis of sulfa drugs, the Ministry of Chemical Industry again raised the demand for isopropyl alcohol. Briefly, studies of the key step, absorption of propylene in acid (87%) began in the laboratory with the following scouting runs: bubbling finely divided gas through acid at atmospheric pressure; passing the gases into the acid with violent agitation; the same, in the presence of kerosene. Of these, the last alone gave yields approaching 1 mole of alcohol per mole of sulfuric acid (after hydrolysis and distillation), based on three hour runs. In a steel tower (about 10' high) packed with steel rings, over which a charge of acid was recirculated, propylene was absorbed from a stream of gas at 5-10 atm. to give 1 mol/mol in 3 hours. Yields based on propylene were

inversely proportional to the rate of gas flow. In a different type of scouting experiment, 87% sulfuric acid was shaken in a steel bomb with a liquid C_3 fraction containing 30-40% propylene. Yields of 1 mol/mol were obtained after 15-30 minutes at 15-20°C, and yields of 1.6 mol/mol after 2 hours. Substantial amounts of diisopropyl sulfate were collected when the hydrocarbon phase was separated from the sulfuric acid and allowed to evaporate. Hydrolysis of this diester gave 80% of the theoretical alcohol, 20% apparently going off as propylene.

16. "Based on data from these batch experiments a pilot plant was designed as shown in the sketch:



17. "Liquid C_3 fraction and 87% sulfuric acid are fed continuously into a water jacketed autoclave. A portion of the reaction mixture passes to a settler. From this, hydrocarbon phase may be passed at a controlled rate to a hot-water heated flash evaporator for separation of diisopropyl sulfate; acid phase is likewise withdrawn continuously; any excess of one or the other phase may be recycled from the settler back into the autoclave.

18. "Results from this unit were not available when I left in April 1952.

19. "Another project of considerable interest was the oxidation of paraffin to fatty acids for use in a replacement soap. (Irimescu, Vladianu) The same problem was being attacked by ICECHIM (research institute of the chemical industry's ministry) and Sovrompetrol. In this race to eliminate a source of universal complaint among the people and thus gain political prestige, these laboratories jealously protected their know-how and would not coordinate their efforts beyond an agreement that each study the use of a different starting material. ICECHIM used commercial paraffin wax. ICF used topped paraffinic crude ('petrolatum' so-called). Technically all of these groups were simply attempting to copy the process used by the Germans 10 or 15 years ago. Molten paraffin was bubbled with air at essentially atmospheric pressure, 130-140°C., with a trace of permanganate as an initiator. After about a year in the lab Irimescu passed to a pilot plant study using an aluminum tower about 6" diameter and 8 or 10' high. No reliable analyses for oxygenated (hydroxy, or keto) acids in the product were shown, nor was a satisfactory procedure for working up the product into a hard oil-free soap developed. ICECHIM made a hard, dark-brown bar of oil-free soap, of rather poor lathering qualities, by neutralizing the oxidized paraffin and passing the entire mixture of soap, water and neutral oil through a pipe-still. By flash distillation at 200°C the water and oil went overhead leaving the soap. The equipment and operating costs of such a process would obviously make it a poor competitor for laundry soap, pricewise.

20. "Another process studied intensively for a time was catalytic cracking. The objective of the studies was to determine whether catalysts of standard activity could be produced from indigenous materials. Both activated clays and synthetic cats were tested; laboratory values comparable to figures cited for US plant yields were obtained. Only one catalyst was run for more than a few hours. After 24 hours on gas-oil cycle (20 minute periods, followed by 20 minutes regeneration with air) its activity had dropped to give 25% gasoline instead of the initial 37%. Examination of the pellets showed clearly visible sintering on some surfaces. Since a report of these tests using a more controlled regeneration was not [illegible] no data on whether a catalyst with long life was actually produced.

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21. "Interest in this project dwindled considerably from the vigorous beginning in winter 1950-1 when it was assigned 3 full time chemists - engineers, 2 technicians, and 2 part-time PhD's (Ion Nicholescu, for the chemical studies, and Ion Popescu, for the minerolcgical work on clays). Six months later, interest had reached the point where the director made no inquiry during an entire month in which the only reactor stood broken down and out of commission. It may be added that no coordination was observable with other groups; no indications were ever received whether a plant had been definitely decided on, whether a process (Houdry fixed-bed, Thermofor or fluid) had been selected, or whether any engineering data were desired.

22. "Among other projects, of still lesser importance, the following may be listed:

Preparation of pharmaceutical grade petrolatum (Dr. Ion Segal, formerly of Socony-Vacuum).

Recovery of oil from bituminous sands and clays using hot water or alkaline solutions (Dr. Ion Robu, of Astra-Romana, and Dr. Radu Tunescu).

Recovery of sulfates as sodium sulfate and bisulfate from acid sludges (Robu and others).

Blowing of asphalts. (Tabachnik, of Astra-Romana).

Classification of crude oil produced by several thousand individual wells, based on distillation and inspection data. (4-5 chemists).

Recovery of clay and/or oil values from spent decolorizing clays.

Oxidation inhibitors for lube oils.

23. "Any estimate of petroleum research activity in Rumania should bear in mind the following points:

a. Applied industrial research and development is a new thing there. Philosophy, procedures, experienced personnel and physical equipment cannot be created overnight.

b. The present economic situation severely limits not only the acquisition of scientific materials, such as must be imported from Germany, the USSR or other countries, but even makes extremely difficult the purchase of ordinary steel pipe, valves, and many common chemicals. This is all compounded by the severe gasoline rationing, the importance of which only becomes clear when one realizes that materials or services are obtained only by on-the-spot negotiation with friends or connections, since no one will admit by phone or through official channels, that he can be of any help.

c. Morale is in a lassitude verging at times on sabotage. This is due to many well known factors, such as poor living conditions, lack of confidence in the Government and the technical directors, and the fact that most of the projects being self-assigned, it is speculative whether any project is observed from above unless a special priority is given, or whether the results of any study are likely to ever be put into application."

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